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## ON THE USE OF STANDARD GLASS POWDERS IN REFRACTIVE INDEX DETERMINATIONS

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The dispersion method, employed for the first time by H. E. Merwin<sup>1</sup> and developed\* by S. Tsuboi,<sup>2</sup> is now used extensively. Any one who has tried this method knows how useful it is for the determination of refractive indices of minute crystals. Later the double variation method was proposed by R. C. Emmons,<sup>3</sup> and his apparatus is now on sale.

For a number of years the present writer has used an apparatus as shown in Fig. 1, which is a modification of Tsuboi's. It consists essentially of three thermostats containing water of different temperatures, and a system of water circulating tubes which connects the thermostats to a disk on the microscope stage. The disk is similar to that used by A. N. Winchell. The temperature on the stage can be quickly controlled by varying the temperature of the circulating water by means of a specially devised cock.

In connection with the dispersion method, or the double variation method, the Abbé refractometer is ordinarily used for determining the refractive indices of the immersion media. Accordingly, the method is not applicable to crystals having higher indices than 1.7. Also, there are certain inconveniences in the usual method.

\* In spite of the opposition from G. Tunell and G. W. Morey (*Am. Mineral.*, Vol. 13, 1932, p. 376) the present writer uses this statement, for S. Tsuboi showed for the first time how the principal refractive indices of double refracting crystals can be determined by the dispersion method. Moreover, Tsuboi proposed the use of Hartmann's dispersion net in the dispersion method, and simplified the procedure of finding graphically the refractive indices of powdered minerals for light of different wave lengths.

<sup>1</sup> H. E. Merwin and E. Posnjak, *J. Amer. Chem. Soc.*, Vol. XLIV, 1922, pp. 1965-1994.

<sup>2</sup> S. S. Tsuboi, *Miner. Mag.*, Vol. 18, 1923, p. 108.

S. S. Tsuboi, *Japanese Journ. Geol. and Geogr.*, Vol. III, 1924, p. 19.

S. S. Tsuboi, *Journ. Geol. Soc. Tokyo*, Vol. XXXII, 1925, pp. 1-6.

S. S. Tsuboi, *Journ. Geol. Soc. Tokyo*, Vol. XXXVII, 1930, p. 39.

<sup>3</sup> A. N. Winchell and R. C. Emmons, *Am. Mineral.*, Vol. XI, 1926, p. 115.

R. C. Emmons, *Am. Mineral.*, Vol. XIII, 1928, p. 504 and XIV, 1929, p. 441.

In the first place, there is often a large difference between the temperature on the microscope stage and that of the Abbé refractometer, which makes the result unreliable, though the correction may be obtained by determining the temperatures with the thermocouple of copper and constantan, as E. H. Ashton and W. C. Taylor<sup>4</sup> have suggested. In the second place, the immersion liquid may partially evaporate and its composition and refractive index may change during the experiment, especially at the higher temperatures. We may overcome this trouble by using pure compounds as immersion media instead of mixtures as pointed out by Winchell and Emmons<sup>5</sup>; but pure liquids with appropriate refractive indices are sometimes very difficult to prepare.

In order to avoid the inconveniences enumerated above, the writer proposes here the use of *standard glass powders* in refractive index determinations with the microscope. By this method the refractive index and dispersion of the immersion liquid can be determined simultaneously with those of the crystal mounted on the microscope stage, without using the Abbé refractometer.

In the method here proposed we have no need of using numerous thermostats and water circulating systems, as it is not necessary to keep the refractometer and the microscope stage at the same temperature. It is only necessary to have such apparatus as will maintain the microscope stage at a constant temperature. An electric heating stage with a device for automatic temperature control, such as an Eisenberg or a Walton stage (Leitz), for instance, will meet this need.

### I. ISOTROPIC CRYSTAL

Immerse the crystal under investigation in liquid  $X$  with proper standard glass powders,  $A$  and  $B$ , and the refractive index of the crystal as well as those of the glass powders are compared with that of the liquid, using a petrographic microscope and a Fuess monochromater. The refractive indices and dispersions of the standard glasses must have been determined previously and plotted on a Hartmann's dispersion net (Fig. 3). Suppose the refractive index of the liquid  $X$  matches those of the glass powders  $A$  and  $B$ , respectively, at wave lengths  $a$  and  $b$ . Then, locate the points  $X_a$  and  $X_b$  (Fig. 2) corresponding to  $a$  and  $b$ , on the disper-

<sup>4</sup> E. W. Ashton and T. C. Taylor, *Am. Mineral.*, Vol. XIII, 1928, p. 411.

<sup>5</sup> *Op. cit.*



sion lines  $A$  and  $B$ ; the connecting line  $X_aX_b$  represents the refractive index and dispersion of the liquid  $X$ .

Further procedure is quite similar. The wave length  $x$ , for which

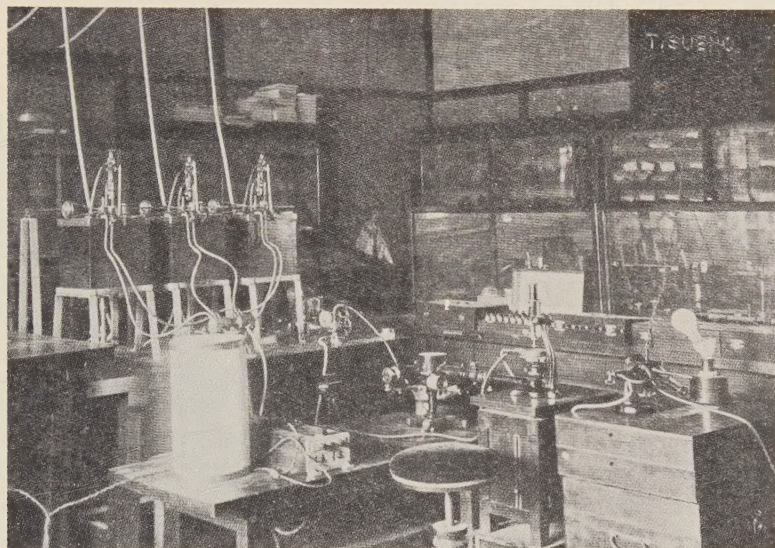


FIG. 1

TABLE I

	Li	Na	Tl		Li	Na	Tl
No. 1	1.6690	1.6764	1.6834	No. 16	1.7178	1.7263	1.7344
2	1.6715	1.6790	1.6862	17	1.7212	1.7299	1.7382
3	1.6740	1.6815	1.6887	18	1.7253	1.7342	1.7424
4	1.6770	1.6847	1.6919	19	1.7296	1.7383	1.7467
5	1.6804	1.6879	1.6950	20	1.7337	1.7425	1.7510
6	1.6830	1.6906	1.6977	21	1.7382	1.7472	1.7556
7	1.6867	1.6943	1.7016	22	1.7429	1.7520	1.7606
8	1.6902	1.6980	1.7052	23	1.7473	1.7566	1.7654
9	1.6936	1.7016	1.7090	24	1.7506	1.7599	1.7689
10	1.6977	1.7056	1.7132	25	1.7540	1.7636	1.7724
11	1.7018	1.7099	1.7174	26	1.7581	1.7681	1.7771
12	1.7038	1.7120	1.7198	27	1.7642	1.7741	1.7834
13	1.7078	1.7160	1.7239	28	1.7690	1.7790	1.7886
14	1.7116	1.7202	1.7281	29	1.7744	1.7847	1.7944
15	1.7152	1.7239	1.7319	30	1.7776	1.7881	1.7979

the refractive index of the crystal matches that of the liquid  $X$ , is determined. Then, the experiment is repeated either with the same liquid at another temperature, or with another liquid,  $Y$ , and the wave length  $y$  determined, at which the matching occurs. To obtain  $n_D$ , join the points  $n_x n_y$  corresponding to  $x$  and  $y$ , respectively, to the dispersion lines of  $X$  and  $Y$ . The connecting line  $n_x n_y$  represents the refractive index and dispersion of the crystal, and the intersection of this line with the  $D$  line will give  $n_D$ .

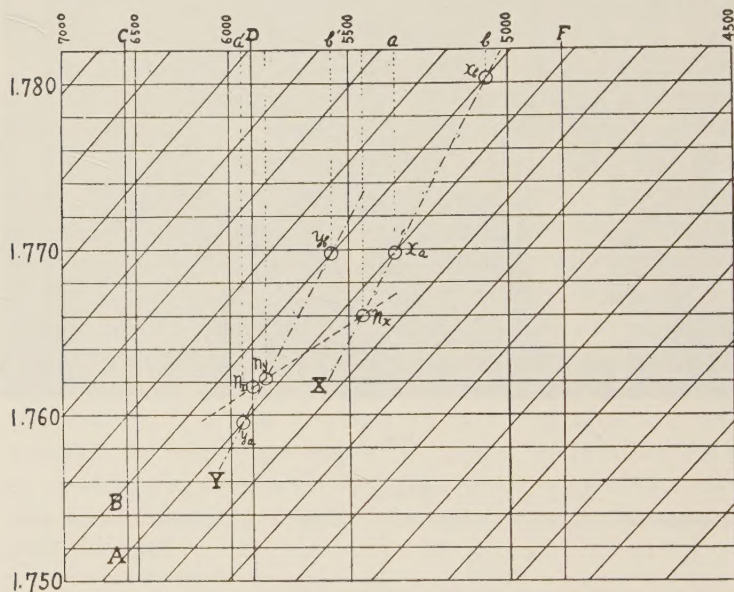


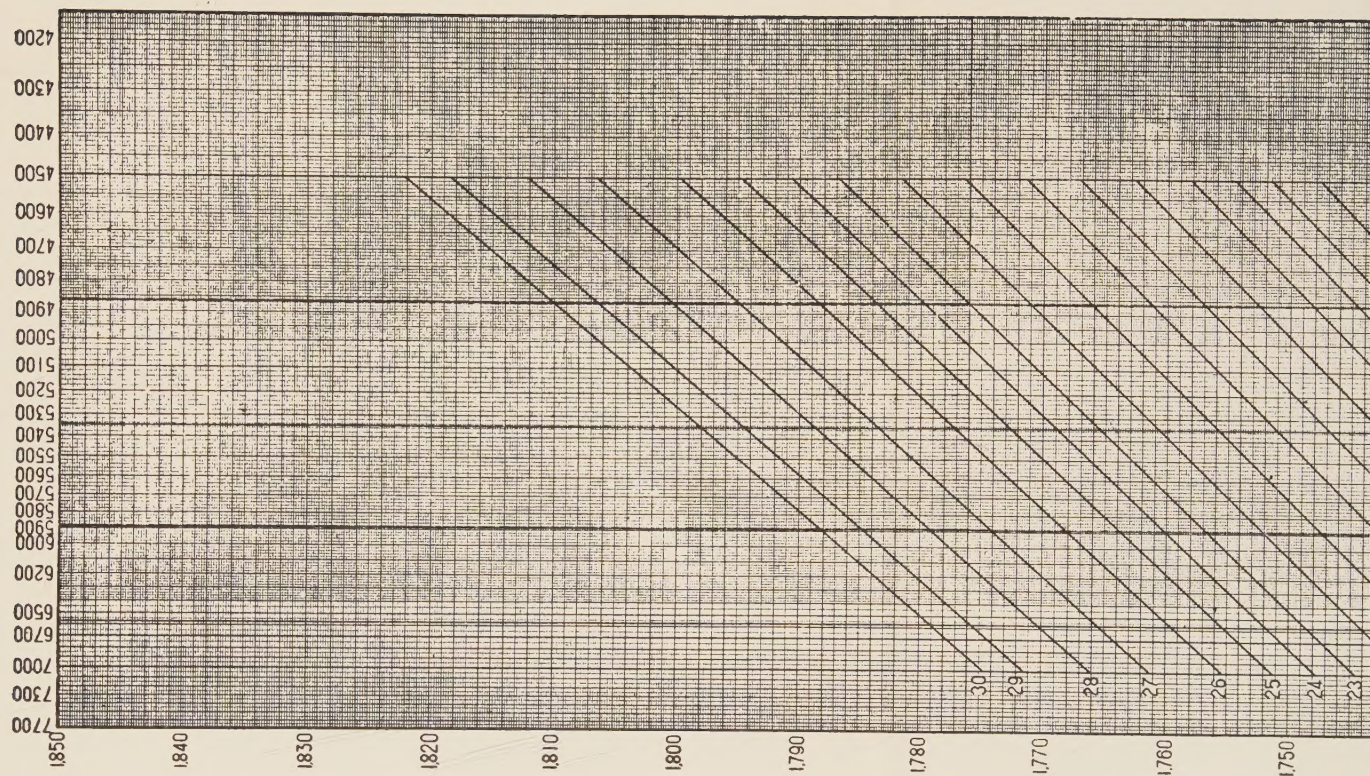
FIG. 2

## II. UNIAXIAL CRYSTAL<sup>6</sup>

In order to find the  $\omega$  and  $\epsilon$  indices of a uniaxial crystal, it is best to follow the method outlined by S. Tsuboi. The following example will suffice to demonstrate that method. The powders of a crystal of corundum and standard glasses, Nos. 23 and 24 whose dispersions are represented in Fig. 3, were immersed in a suitable liquid. Examinations were made in monochromatic light to determine at which wave lengths the refractive indices of the standard glasses and  $n_1$  and  $n_2$  of each powdered grain of mineral would

<sup>6</sup> The following procedures are quite similar to those described in S. Tsuboi's paper, *Journ. Geol. Soc. Tokyo*, Vol. XXXII, 1925, pp. 2-6.







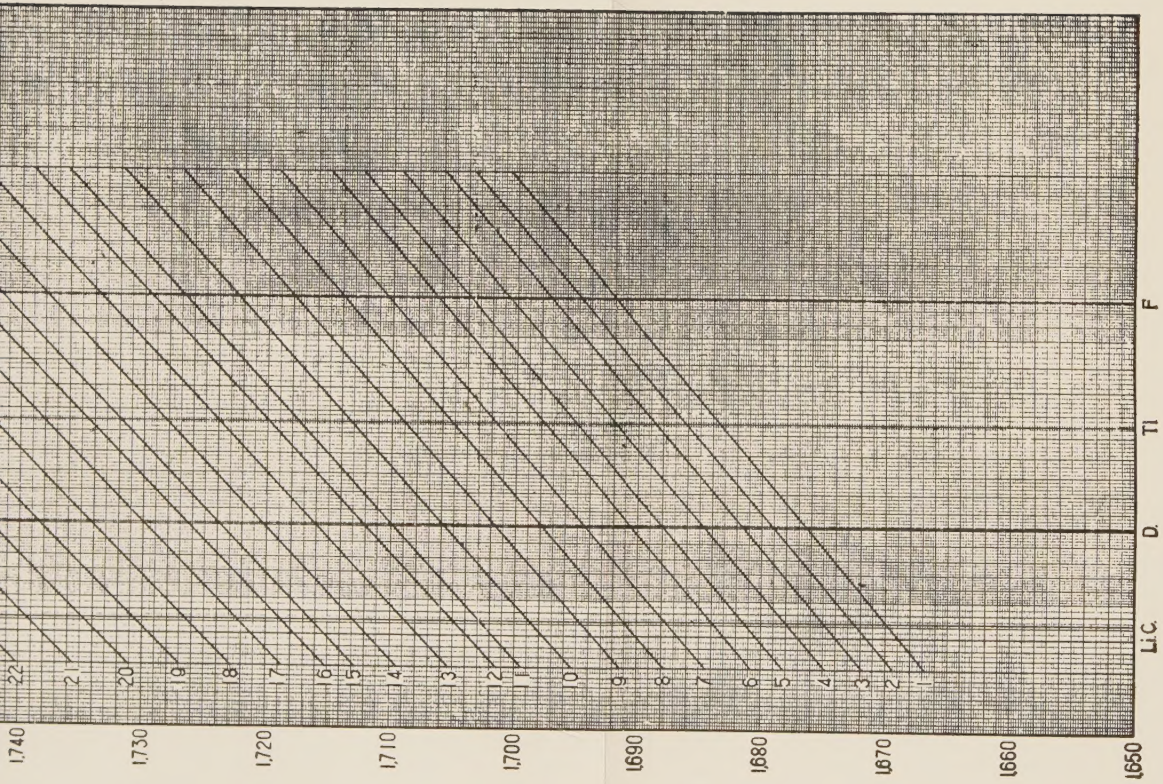


Fig. 3

TABLE 2

Standard glasses Liquid	No. 23 550 $\mu\mu$	No. 24 490 $\mu\mu$
Corundum	$n_1$	$n_2$
	525 $\mu\mu$	495 $\mu\mu$
	521	494
	513	495
	510	495
	530	494
	511	495
	522	495
	513	495
	509	494
	497	495
	504	495
	520	495
	515	495
	542	494
	520	495
	500	495

TABLE 3

Standard glasses Liquid	No. 23 604 $\mu\mu$	No. 24 545 $\mu\mu$
Corundum	$n_1$	$n_2$
	533 $\mu\mu$	513 $\mu\mu$
	529	513
	535	513
	541	513
	563	513
	545	512
	563	513
	543	513
	556	512
	535	512
	547	513
	552	513
	553	513
	525	513
	557	513

TABLE 4

Standard glasses Liquid	No. 24 572 $\mu\mu$	No. 25 513 $\mu\mu$
Corundum	$n_1$	$n_2$
	546 $\mu\mu$	522 $\mu\mu$
	555	520
	574	522
	544	523
	572	523
	546	522
	550	520
	533	522
	532	520
	527	522
	546	521
	552	522
	557	522
	545	522
	549	522
	540	522

TABLE 5

Standard glasses Liquid	No. 7 514 $\mu\mu$	No. 8 478 $\mu\mu$
Hyper- sthene	$n_1$	$n_2$
	506 $\mu\mu$	480 $\mu\mu$
	538	violet
	536	475
	506	477
	518	485
	518	violet
	500	violet
	496	violet
	500	475
	513	472
	502	violet
	527	473
	497	470
	547	477
	529	violet



match that of the liquid. The observations were made on 15 grains of the mineral chosen at random and it was found that the matching occurred for the wave lengths given in Table 2.

The experiment was then repeated at another temperature, or by immersing the powders in another liquid, and the results as shown in Table 3 were obtained.

To find the dispersion of the liquid, mark the points corresponding to  $550\ \mu\mu$  and  $490\ \mu\mu$ , respectively, on the lines Nos. 13 and 14, and connect them.

As can be seen in the above tables the value for  $n_2$  is constant in every grain, whatever its orientation. This corresponds to that of  $\omega$ .

To obtain  $\omega_D$  mark the points corresponding to  $495\ \mu\mu$  and  $513\ \mu\mu$  (the mean of the wave lengths in the column for  $n_2$  in Tables 2 and 3) on the lines representing the first and second liquids in Fig. 4 and connect them; then the intersection of this line with the  $D$  line will give  $\omega_D$  of the mineral.

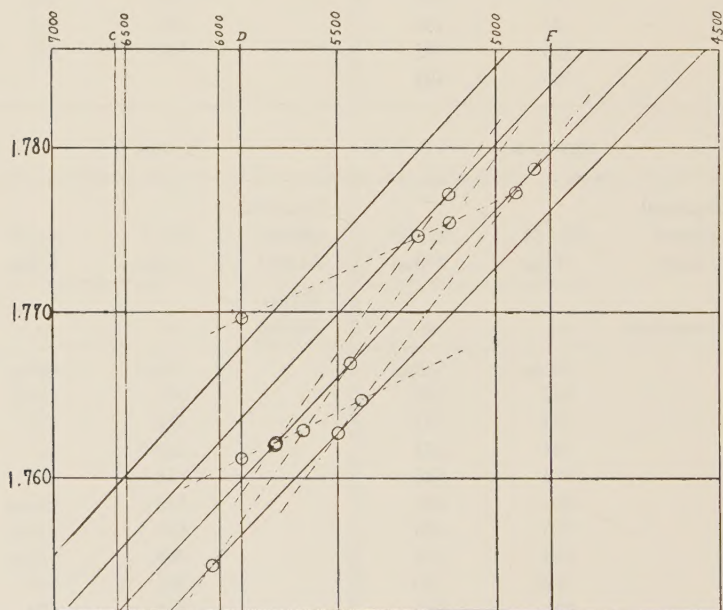


FIG. 4

The value of  $n_1$  observed in each grain is that for the extraordinary ray, and varies from  $\omega$  to  $\epsilon$ ,  $\epsilon$  being, in this case, the minimum value of  $n_1$ . Of the various values for  $n_1$  given in Table 2, the value



for the light of wave length  $542\ \mu\mu$  (the longest wave length) is the lowest, since the dispersion of the immersion liquid is stronger than that of the crystal. Likewise, of the values of  $n_1$  in Table 3,  $563\ \mu\mu$  is the lowest. Assuming that these lowest values of  $n_1$  represent approximately  $\epsilon$ ,  $\epsilon_D$  can be obtained graphically in a manner similar to that indicated for  $\omega_D$ .

The values of  $\omega_D$  and  $\epsilon_D$  thus obtained are:

$$\omega_D = 1.769$$

$$\epsilon_D = 1.761$$

And the dispersion is:  $\omega_F - \omega_C = 0.013$

### III. BIAXIAL CRYSTAL

To demonstrate the method for obtaining  $\alpha_D$ ,  $\beta_D$ , and  $\gamma_D$  of biaxial crystals, a hypersthene crystal from Taihoku, Taiwan, Japan, will be taken as an illustration. The powders of a hypersthene crystal and standard glasses Nos. 7 and 8, whose dispersions are represented in Fig. 3, were immersed in liquids, and the refractive indices of the mineral, standard glasses and liquids were compared in the same way as in the case of the corundum. The results are given in Tables 5, 6, and 7.

TABLE 6

Standard glasses	No. 7	No. 8
Liquid	$580\mu\mu$	$526\mu\mu$
Hyper- sthene	$n_1$	$n_2$
	$564\mu\mu$	$503\mu\mu$
	531	509
	587	499
	570	509
	540	526
	533	517
	537	518
	572	506
	527	500
	587	503
	545	491
	529	495
	578	502
	537	515
	528	509

TABLE 7

Standard glasses	No. 9	No. 10
Liquid	$610\mu\mu$	$537\mu\mu$
Hyper- sthene	$n_1$	$n_2$
	red	$559\mu\mu$
	$625\mu\mu$	565
	610	558
	red	576
	red	560
	red	554
	red	565
	red	549
	638	570
	red	553
	red	557
	red	580
	red	609
	613	567
	red	566

S. Tsuboi has shown the following relationship  $\alpha \leq n_1 \leq \beta \leq n_2 \leq \gamma$  holds for a grain of a biaxial crystal of any orientation. Because of this relationship it is clear that the refractive indices of the mineral that match  $n$  of the first liquid for light of wave lengths 547  $\mu\mu$ , and 496  $\mu\mu$ , as well as those that match  $n$  of the second liquid for 587  $\mu\mu$ , and 527  $\mu\mu$ , are  $\alpha$ , and  $\beta$  respectively. And the refractive index that matches  $n$  of the second and the third liquids, respectively, for 526  $\mu\mu$ , and 553  $\mu\mu$  corresponds to  $\gamma$ . Marking the points representing these wave lengths on the line of liquids in Fig. 5 and

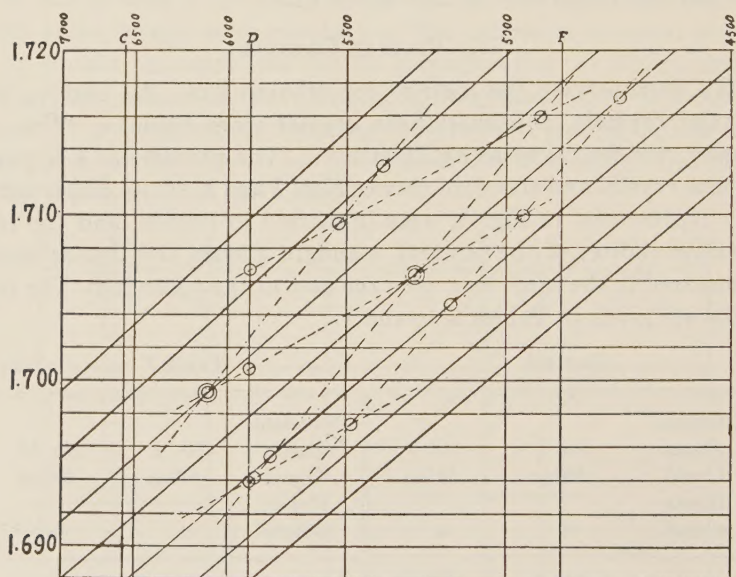


FIG. 5

applying the same graphical method as in the preceding examples, the following values were obtained:

$$\alpha_D = 1.694; \beta_D = 1.701; \gamma_D = 1.707; \beta_F = \beta_C = 0.015.$$

#### METHOD FOR PREPARING THE STANDARD GLASS

Any type of colorless and transparent glass with appropriate refractive index and dispersion, which is stable and not easily devitrified, may meet the need of a standard glass, without regard to its composition. However, a glass of weak dispersion is preferred, because then the difference in dispersion between the glass and



the immersion liquid is large and a comparatively small number of standard glasses are sufficient for our purpose.

The refractive indices and dispersions of the glasses prepared for the present purpose are shown in Table I, and are represented on Hartmann's net (Fig. 3).

The process of making a standard glass is as follows. At first two kinds of glasses of the following extreme compositions, *A* and *B*, are made in the glass furnace by mixing  $\text{As}_2\text{O}_3$ , quartz sand,  $\text{Pb}_3\text{O}_4$  and  $\text{KNO}_3$ .

	Quartz	$\text{As}_2\text{O}_3$	$\text{PbO}$	$\text{K}_2\text{O}$	
A	38.0	0.3	56.7	5.0	(%)
B	23.0	0.3	75.0	1.7	(%)

When the mixture is heated caution must be taken against blackening due to the separation of metallic lead caused by the action of the reducing flame when used for a long period. The use of  $\text{K}_2\text{CO}_3$  is avoided, as it may liberate carbon dioxide which sometimes reduces the lead oxides. For melting the glass a crucible of chamotte is used and it is heated for about two hours at about  $1300^\circ\text{C}$ . When the molten glass becomes free from bubbles, it is immersed into water and a frit obtained. The frit is crushed in a ball mill to a fine powder and sieved through 4900 mesh. It is important to render the powder extremely fine, for otherwise it may be difficult to obtain a homogeneous glass in the second heating.

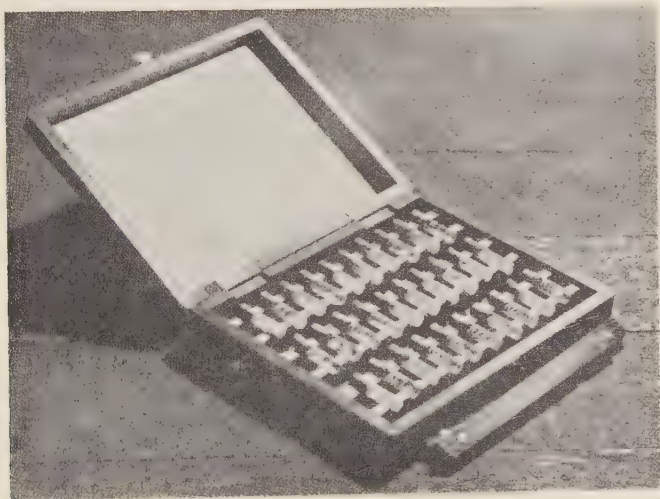


FIG. 6

Next the glass powders of the extreme compositions *A* and *B* are mixed in various proportions and a series of mixtures, 50 grams each, obtained. These are melted in a carbon resistance electric furnace, keeping the preparations at 1000°–1200°C, for about an hour and a half, stirring the melt from time to time with a small platinum spatula attached to the end of an iron rod in order to obtain a homogeneous melt. A small amount of arsenic and antimony oxides are added to facilitate and to expedite the refining of the glass. The crucible used is made chiefly of alumina—e.g., “S.M.” of Samposha Company. After removing the crucible from the furnace, it is placed in a small dish of chamotte and cooled to room temperature in about 3 hours. The internal stresses produced upon cooling the glass causes no harm for our purpose, and there is little need for annealing. The outermost part, about 3 mm. thick, is removed, and the homogeneity of the remaining central part is ascertained by comparing the refractive indices of different portions of the mass with one another by the dispersion method. The refractive index and dispersion of the standard glass thus obtained are determined by the prism method. Table I shows the indices and dispersions of the standard glasses manufactured for the present purpose, and Fig. 3 represents the dispersion diagrams plotted on Hartmann’s net.

#### ACKNOWLEDGMENTS

The writer wishes to thank Prof. S. Tsuboi of the Imperial University of Tokyo for his suggestions and criticisms during the preparation of this paper. In the manufacture of the standard glasses the writer was greatly assisted by Mr. T. Echizenya.



## SULFOHALITE AND OTHER MINERALS FROM THE OTJIWALUNDO SALT PAN, SOUTH WEST AFRICA

WILLIAM F. FOSHAG,<sup>1</sup> *United States National Museum.*

The mineral sulfohalite was first described by Hidden and Mackintosh<sup>2</sup> as a chlorosulfate of soda from Searles Lake, California. They record at that time but three known specimens; well formed dodecahedrons reaching 3 cm. in diameter associated with hank-site. The finest of these is now in the Bement Collection of the American Museum, another is in the British Museum and a portion of the third crystal in the Brush Collection at Yale. Later, Penfield<sup>3</sup> reinvestigated the chemical composition of the mineral, using material from the crystal in the Brush Collection, and found it to be a compound of the formula:  $2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$ .

Two octahedral crystals were later obtained by Mr. Hoyt S. Gale from Dr. S. P. Sadtler of Philadelphia, one of which was consumed in making an analysis<sup>4</sup> and the second was deposited in the United States National Museum. Since that time this writer has found a few additional crystals in material from Searles Lake, the largest crystal being an octahedron 1.5 cm. across. A considerable quantity of Searles Lake salts from deep well borings have been examined for rare minerals and the conclusion has been reached that sulfohalite is one of the rarest of the species found there.

It is with considerable interest, therefore, to record a new locality for sulfohalite, at Otjiwalundo Salt Pan, S.W. Africa, where to judge from the specimens available it occurs in fair abundance. These specimens were obtained by Mr. George L. English during a collecting trip in South West Africa. According to Mr. English the Otjiwalundo Salt Pan lies west of the great Etosha Pan and about 80 kilometers northwest of Okaukuejo, the police station on the southwest corner of the Etosha. It is about 250 kilometers north of west of Otavi.

The specimens of the suite of minerals from this locality contain trona with sulfohalite, thenardite, both single and twinned crystals, and thenardite with pirssonite.

<sup>1</sup> Published by permission of the Secretary of the Smithsonian Institution.

<sup>2</sup> *Am. Jour. Sci.*, **36**, 463, 1888.

<sup>3</sup> *Am. Jour. Sci.*, **9**, 425, 1900.

<sup>4</sup> Gale and Hicks: *Am. Jour. Sci.* **38**, 273, 1914.

## TRONA

The trona specimens consist of broad blades, coarsely reticulated to form an open mesh of crystals. The individual crystals are rounded and except for their unusually large size are of little special interest. Crystals 8 centimeters long and 3 centimeters wide were observed on some of the specimens. With the trona are associated the crystals of sulfohalite and occasionally poor crystals of thenardite.

## SULFOHALITE

If one can judge from the sample at hand this rare chloride, fluoride and sulfate of sodium is a common mineral in the Otjiwalundo Salt Pan. In some of the specimens the sulfohalite, although irregular in shape, reach a size of 2 centimeters and is abundantly distributed through the coarsely reticulated trona blades. The smaller crystals are dodecahedral in habit but somewhat distorted. The larger crystals are also dodecahedral but are usually aggregated into roughly parallel groups.

Like the sulfohalite from Searles Lake, the larger crystals have a yellowish cast, or where they inclose muds, a pale gray color. Under the microscope the mineral is isotropic with an index of refraction of 1.455.

An analysis of this mineral gave the following results:

SULFOHALITE, OTJIWALUNDO SALT PAN, S.W. AFRICA

Na	36.83	F	4.95
SO <sub>4</sub>	48.90	H <sub>2</sub> O	0.15
Cl	9.20	Insol.	0.50
			100.53

The results of this analysis agree well with those found by S. L. Penfield and W. B. Hicks on the Searles Lake mineral and with the theoretical values for the formula:  $2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$ .

## PIRSSONITE

Pirssonite is a rare double carbonate of calcium and sodium previously found only at Searles Lake. Here this mineral is fairly abundant in certain layers of the playa associated with halite, hanksite, trona, northupite, gaylussite and borax. Crystals reaching a centimeter are unusual and imperfect in development, but smaller ones are abundant in some samples, and present a variety of crystal



habits. Since this mineral is recovered entirely from well drillings, it is not widely distributed in collections, but samples from several scattered parts of the playa of Searles Lake indicate that the mineral is abundant.

On the samples from the Otjiwalundo Salt Pan, pirssonite is only sparingly present but it is associated in such a manner as to suggest that the larger crystals of thenardite and trona grew in a sand of quartz grains and pirssonite crystals. The mud adhering to the trona specimens contained a number of clear but minute crystals. Larger crystals were found in the sandy material attached to or in pockets of a large thenardite crystal. The largest of these pirssonite crystals seldom exceed a millimeter in length and when not distorted have a hexagonal outline. Many of them, however, are elongated along the  $a$  or the  $c$  axis or even transversely, so that it is usually necessary to mount a crystal several times before the proper orientation is obtained. The measured crystals show the common forms:  $b$  (010),  $m$  (110) and  $p$  (111). The measurements are given in the following table:

MEASUREMENTS OF PIRSSONITE FROM OTJIWALUNDO SALT PAN

Form	$\phi$	$\rho$	Face
$p$	$60^{\circ}32'$	$31^{\circ}37'$	Medium
$b$	0	90	Large
$m$	$60^{\circ}35'$	90	Small

Their habit is similar to one of the common types from Searles Lake in which the brachypinacoid is broad and the unit pyramid and the prism bevel the edges (Fig. 2).

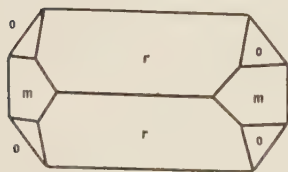


FIG. 1

FIG. 1. Thenardite from the Otjiwalundo Salt Pan. S. W. Africa.

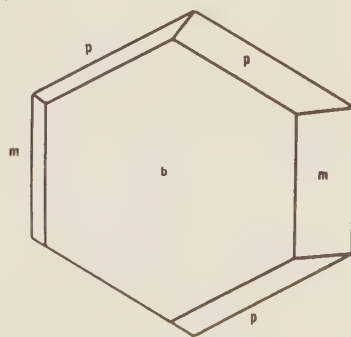


FIG. 2

FIG. 2. Pirssonite from the Otjiwalundo Salt Pan.

The following optical properties confirm the crystallographical determination: Biaxial, positive with medium optic angle. Dispersion distinct with  $\rho$  less than  $\nu$ .  $\alpha = 1.504$ ,  $\beta = 1.509$ ,  $\gamma = 1.575$ .

#### THENARDITE

Two specimens of thenardite were found in the minerals studied, a large group of crystals with no associated minerals other than a few rough crystals of salt, and a large crystal with attached trona and small nests of sand and pirssonite.

The large crystal is 12 centimeters long by 9 centimeters wide and shows the common forms  $r$  (101),  $o$  (111) and  $m$  (110). The habit of this crystal differs from any of those shown in Goldschmidt's Atlas in that the macrodome  $r$  (101) is by far the most prominent face, the unit pyramid and prism being reduced to modifying faces (Fig. 1).

The large group consists of a mat of small simple crystals of a habit similar to the large one (Fig. 1) over which are scattered large twins up to 5 centimeters long. These twins are made up of crystals with forms similar to the simple crystals twinned on the face  $r$  (101), giving a habit somewhat similar to the "butterfly" twins of gypsum.

In a modification of this habit, shown only in a few of the smaller twins, the prism is practically missing and the outer face  $r$  is elongated so that the crystal is rectangular and modified by small grooved terminations. These are similar to many other twins where a marked flattening of the twinned crystals is a common feature.



## A LARGE MONAZITE CRYSTAL FROM NORTH CAROLINA<sup>1</sup>

WALDEMAR T. SCHALLER, *U. S. Geological Survey.*

In a recent number of this journal Palache<sup>2</sup> has given the dimensions of a number of large crystals of various minerals and has asked for information concerning others. The largest crystal of monazite (nearly a foot high) known to the writer is here described, with some notes on the occurrence. It is hoped that others, as requested by Palache, will place on record what information they have on the largest known crystals of the different mineral species so that eventually the compilation will be fairly complete and have considerable value.

For many years monazite has been known to occur about 15 miles north of Asheville, N. C. Many of the crystals found are of unusual size, those as large as one's hand being rather abundant. The large crystal here described may be the "large rough crystal that weighed almost exactly 60 pounds" mentioned by Pratt.<sup>3</sup> The occurrence is generally known as Mars Hill, being in Madison County, N. C.,  $5\frac{1}{2}$  miles due east of Marshall, 3 miles southwest of Mars Hill, midway between Sexton and Outlook, on the road from Halewood to Jupiter, a mile north of where the road, going south, first abuts White Oak Creek. The deposit is on the farm of the late Rev. N. P. M. Corn who very graciously afforded the writer all opportunities for studying the occurrence.

The locality was visited in 1918 as part of the War program on conservation of domestic mineral supplies. The large crystal here described was then in the possession of Mr. Corn and has only recently been acquired by Mr. Burnham S. Colburn of Biltmore Forest, N. C., who very kindly has permitted the writer to publish this note. As far as known it is the largest crystal of monazite preserved in any collection. It is shown in Fig. 1. It measures  $6\frac{1}{2}$  inches ( $18\frac{1}{2}$  centimeters) along the *a*-axis,  $9\frac{1}{2}$  inches (26 centimeters) along the *b*-axis, and 11 inches ( $31\frac{1}{2}$  centimeters) along the *c*-axis. Its present weight is  $58\frac{3}{4}$  pounds. Cleavage fragments break

<sup>1</sup> Published by permission of the Director, U. S. Geological Survey.

<sup>2</sup> Palache, Charles, The largest crystal: *Am. Mineralogist*, 17, pp. 362-363, 1932.

<sup>3</sup> Pratt, J. H., New occurrences of monazite in North Carolina: *Journ. Elisha Mitchell Sci. Soc.*, 28, pp. 153-156, 1913. Zircon, monazite and other minerals, etc., *North Carolina Geol. and Econ. Survey, Bull.* 25, p. 47, 1916.

off very easily and the crystal may have originally weighed somewhat more. The crystal is incomplete and is bounded on one side (the left vertical side in Fig. 1) by a fairly smooth cleavage face of  $m'''$  ( $\bar{1}\bar{1}0$ ), showing the traces of many parallel cleavages parallel to the other face ( $m(110)$ ) of the unit prism and to the base  $c(001)$ . There are also indications of a cleavage parallel to the face of  $v(11\bar{1})$ . The faces of the large crystal are uneven and the entire crystal is somewhat warped, but the measured interfacial angles serve to identify the crystal forms. In further confirmation of the orientation as given, a thin section was cut from a small piece cleaved off parallel to the base  $c(001)$ . This section showed the emergence of the acute bisectrix with the axial plane diagonal to the two cleavages  $m$  and  $m'''$ . The axial plane is normal to  $b(010)$ .

The large front face shown in Fig. 1 is  $m(110)$  with the negative pyramid  $v(11\bar{1})$  below it. The faces on the right are  $b(010)$  and  $c(011)$ . The large cleavage face on the left, perpendicular to the page and therefore not showing, is  $m'''$  ( $\bar{1}\bar{1}0$ ). Small cleavages parallel to  $c(001)$  may be seen in the upper left corner. The crystal evidently was originally much larger and may have weighed nearly a hundred pounds.



FIG. 1.—Large crystal of monazite from near Mars Hill, Madison County, North Carolina. The large face is  $m(110)$ . The scale is in inches.

The crystal of monazite was obtained from a pegmatite dike, striking NE-SW, inclosed in Cranberry granite containing parallel tongues of Carolina gneiss. According to local report, the deposit of monazite was discovered in 1902 by Paul S. Corn and limited operations continued for two years, several large crystals and masses being obtained. About half way up the hillside a shaft 45 feet deep was sunk by Mr. F. M. Salyer of Johnson City, Tenn., and monazite was reported as found all the way. Early in 1918, a six foot deep open cut and several small prospect holes were dug by Mr. Gitt of Roanoke, Va.

Several hundred pounds of crystals and of cleavage pieces have been carried away by visitors and in 1918 a large box on the property still contained several hundred pounds of such specimens, many of which were as large as one's hand. Possibly a ton or so of monazite was obtained altogether.

The rocks on the farm are much decomposed but seem to be largely Cranberry granite. Most of the rocks in the open cut have been covered with loose dirt but the presence of a pegmatite, at least several feet wide, could be seen. In addition to at least one distinct pegmatite dike, there are numerous parallel stringers in the adjoining rock, parts of which are so pegmatized that on decomposition the lighter color of the pegmatite stringers is about the only criterion of their former presence. Masses of decomposed feldspar, of massive quartz and of compact dark green mica, each of which is several feet thick, testify to the presence of at least one definite pegmatite dike of workable size. The determined length of the pegmatite is not over a few feet although it presumably extends southward for several hundred feet over the brow of the hill, as monazite cleavage fragments and imperfect crystals have been found abundantly on the eastern slope of the hill, at least that distance from the open cut. No exposures of the pegmatite were seen on the brow of the hill.

The entire pegmatite zone is not over 15 feet wide and is probably much less. The dike is said to have dipped slightly to the northwest from the vertical. If the open cut paralleled the dike, as seems probable, then its strike was about N 30° E, about parallel to the contact of the tongues of Carolina gneiss with the Cranberry granite. The eastern side of the pegmatite, or at least parts of it, for about a foot in width, is composed essentially of dark green mica. Typical massive quartz, one to two feet thick, adjoins the



green mica. The remainder of the pegmatite apparently consisted chiefly of feldspar, now decomposed.

In the mica seam is a zone, averaging from one to four inches in width but locally becoming much thicker, which is very rich in monazite and which has furnished much of the monazite obtained from this locality. There may have been more than one such zone rich in monazite in the mica seam. It also seems probable that there were several lens-like zones rich in monazite rather than a single continuous zone but the available exposure of only a few feet did not permit of a definite determination of the character of these zones.

The pegmatite contains monazite outside of the mica seam but not in quantity. A few isolated crystals of monazite, not larger than a pea, were dug out of the decomposed feldspar and also out of the entire mica seam but none was found in the massive quartz.

At the time the deposit was visited (1918) the writer did not appreciate the genetic significance of determining the character of the feldspar, particularly the "decomposed feldspar" containing the small size crystals of monazite, so that it is not known if this feldspar was microcline or an albitic plagioclase. It may be noted however that the immediate matrix of most of the monazite found was a dark green mica.

Samples were collected of: (1) The zone of pegmatized country rock, that is of rock too decomposed for positive identification, but apparently granite or gneiss with numerous white or light colored pegmatite stringers, not over an inch thick; (2) The white decomposed feldspar; (3) The mica seam across its entire width; (4) The monazite-rich zone in the seam of mica. The samples were taken regularly from areas four inches square, a total of 48 samples being collected. They were mixed and quartered on the ground; the samples, when air dried, weighed about one pound each.

The mica samples were not crushed but were screened (10-mesh) to see if much finely disseminated monazite was present. It was found that nearly all of the monazite present is coarse enough to be caught on a 10-mesh screen, and can readily be picked out by hand.

The samples were then ground and treated with HF and HCl. Trial tests showed that about 20 per cent of the monazite present in the crushed samples was dissolved and lost. The insoluble residue contained besides the monazite, small quantities of ilmenite

and zircon. Their approximate percentage was estimated by microscopic examination and allowed for. The greater part of the monazite present was removed from the samples by the 10-mesh screen, before grinding.

Although the method of procedure was not very exact it served to show the quantity of monazite present in the different samples from which the feasibility of a commercial extraction of monazite can readily be determined.

The results are given below, indicating that only the monazite zone in the mica seam contains any appreciable quantity.

#### PERCENTAGE OF MONAZITE IN SAMPLES

Pegmatized country rock.....	0.05
Decomposed feldspar.....	0.33
Mica seam, coarse <sup>a</sup> .....	0.91
Mica seam, fine <sup>a</sup> .....	0.15
Monazite zone in mica seam, coarse <sup>a</sup> .....	35.04
Monazite zone in mica seam, fine <sup>a</sup> .....	1.86

<sup>a</sup> Coarse is held by a 10-mesh screen and fine passes through.

Although different determinations of the thoria content of the monazite from this locality show some variation, they all agree in showing that at least 5 per cent of thoria, ThO<sub>2</sub>, is present and probably more. Pratt states<sup>4</sup> that a determination showed 5.06 per cent thoria, the writer found 6.06 per cent, another analyst reports 7 per cent, and determinations with the electroscope indicated that some samples contain slightly more.

<sup>4</sup> Pratt, J. H., Zircon, monazite and other minerals, etc.: *North Carolina Geol. & Econ. Survey, Bull.* 25, p. 48, 1916.

# PAULOPOST STILBITE IN THE CAMAS LAND SILL, CHELAN COUNTY, WASHINGTON

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## INTRODUCTION

The Camas Land sill of gabbro and diabase is located in the southern part of Chelan County, Washington, about 10 miles northeast of Blewett Pass and 15 miles west and slightly north of Wenatchee. It is roughly of spoon-like shape, conforming to a synclinal fold the axis of which trends northwest-southeast.<sup>1</sup> The dimensions of the basin-like body are  $3\frac{1}{2} \times 1\frac{3}{4}$  miles and, on account of its shape, the term lopolith may be applicable.<sup>2</sup> From the standpoint of its mode of intrusion it might even be classed as a phacolith. The thickness varies from about 50 feet at one point on the south side to 500 feet or more on the west and northwest. Conformity with the arkosic sandstones and minor conglomerates and shales of the Eocene Swauk formation is imperfect in several places, notably along the northeast margin where the magma of the main body cuts across the sedimentary beds to connect with a thin outlying sheet intruded along a lower horizon. Lack of conformity in the main body is negligible when it is considered that the invaded rock is predominantly massive arkose. The intrusion is thought to have occurred in the Eocene epoch, subsequent to folding and partial erosional beveling of the Swauk formation; it is regarded as one of the manifestations of the vulcanism which gave rise to the Teanaway basaltic flows and dikes occurring extensively south of Mt. Stuart on the southern flanks of the Wenatchee Mountains. Following intrusion of the Camas Land sill, erosion cut deeply into the poorly consolidated sediments, wearing into relief that portion of the area protected by the relatively resistant igneous rock (see Fig. 1). Hence Camas Land is now a topographic eminence bounded by a marginal rampart formed by the upturned edges of the intrusive. It preserves however, a part of the valley of a superimposed stream which successfully carved deep notches in the southern and western parts of the rim before capture of its headwaters by a smaller stream working headward in the readily-yielding sedimentary formation.<sup>3</sup>

<sup>1</sup> Smith, G. O., *U. S. Geol. Survey Geol. Atlas, Mt. Stuart folio*, No. 106, 1904.

<sup>2</sup> Grout, F. F., The lopolith; an igneous form exemplified by the Duluth gabbro: *Am. Jour. Sci.*, 4th ser., Vol. 46, pp. 516-522, 1918.

<sup>3</sup> Smith, G. O., and Curtis, G. C., Camasland; a valley remnant: *Bull. Geol. Soc. Am.*, Vol. II, pp. 217-222, 1900.





FIG. 1. A view (looking northeast) of Camas Land from Tiptop, a prominent peak 4813 feet in elevation which is about 1300 feet above and 2 miles distant from the marginal rim.

The present work on paulopost action and its effects was undertaken on the suggestion of G. E. Goodspeed of the University of Washington. The author gratefully acknowledges his helpful suggestions and criticisms.

#### OUTLINE OF THE PETROGRAPHY

Megascopically the coarse-textured portions of the sill are gray to slightly purple with visible crystals of plagioclase and augite; the fine-textured parts are dark gray to black, commonly showing on the weathered surface a mottling characteristic of many diabbases.

Several closely related rock types are represented in the intrusive body. Gabbro, the predominant rock, makes up approximately 75 per cent; in the order of their abundance, diabase, olivine diabase, and olivine gabbro make up the rest of it. Quartz-bearing modifications occur in those parts of the sill where paulopost action was most prevalent. Plagioclase feldspar, quantitatively the most important mineral constituent, varies from calcic labradorite (An 65, Ab 35) to oligoclase with a preponderance of the former. Zoned crystals within this range of variation are numerous. In the diabase, plagioclase is of early generation with large crystals of augite built around it to form the characteristic diabasic or ophitic texture. In the thicker and coarser-textured portions of the sill, predominately gabbro, the augite crystals average from two to six millimeters in length; the plagioclase averages up to eight millimeters. Magnetite, some of it titaniferous, occurs as

ehedral grains of early crystallization but more of it appears as irregular masses surrounding the feldspar and mafics. Olivine is locally an important mineral constituent, especially near the rarely exposed lower contact and in the small outlier to the northeast of the main intrusive. Approximately 40 per cent of the augite is of the variety pigeonite, distinguishable by its small optic angle. Typical Camas Land gabbro, exclusive of the paulopost minerals, contains 73 per cent plagioclase, 26 per cent augite and pigeonite, 1 per cent magnetite. Both enstatite and hypersthene are present in the sill but are seen in comparatively few sections. With high magnification needles of apatite are seen to cross the non-opaque minerals at random. Quartz when present is associated with stilbite in the interstices. Calcite has a similar occurrence locally where paulopost action was greatest.

The following chemical analysis of the Camas Land gabbro, by Dr. H. N. Stokes, is taken from the folio:

SiO <sub>2</sub> .....	51.98
Al <sub>2</sub> O <sub>3</sub> .....	15.99
Fe <sub>2</sub> O <sub>3</sub> .....	3.10
FeO.....	5.88
MgO.....	5.09
CaO.....	9.68
Na <sub>2</sub> O.....	2.71
K <sub>2</sub> O.....	0.81
H <sub>2</sub> O at 110°.....	0.48
H <sub>2</sub> O above 110°.....	2.08
TiO <sub>2</sub> .....	1.71
P <sub>2</sub> O <sub>5</sub> .....	0.31
MnO.....	0.10
Li <sub>2</sub> O.....	trace
S.....	0.01

#### STILBITE PROPERTIES AND MODE OF OCCURRENCE

The most notable fact brought out by this study is the prevalence of the zeolite mineral stilbite which not only occurs interstitially but likewise penetrates the plagioclase as irregular blebs. This is noted as characteristic of most specimens of the intrusive, the black fine-textured more quickly chilled bottom facies excepted. Stilbite is especially prevalent in those parts of the sill having least thickness and is most common along the northeastern margin. The folio makes no statement of the presence of this mineral but mention is made of an undetermined mineral "probably orthoclase." In thin section the resemblance of the stilbite to

orthoclase is striking, hence, since no orthoclase could be found in a large number of sections, the mineral previously noted was most likely stilbite. The chemical analysis indicates no extraordinary percentage of potash for a gabbro, but it does show the presence of a fairly large quantity of chemically combined water, apparently from stilbite.

In thin section the stilbite appears in irregular masses with wavy extinction; frequently it is in optical units which show good cleavage and nearly parallel extinction. Between crossed nicols its maximum yellow and orange interference colors contrast strongly with the gray and white of the plagioclase. The properties as determined are as follows:  $\alpha=1.487$ ,  $\beta=1.498$ ,  $\gamma=1.50$ ;  $2V=48^\circ$ ;  $X \wedge c=7^\circ$ ; optically negative; good  $\{010\}$  and imperfect  $\{100\}$  cleavages.

#### ORIGIN OF THE STILBITE

The fact that stilbite and small amounts of quartz occur interstitially indicates that both are final products of crystallization. Furthermore, it is apparent that in the last stages of crystallization



FIG. 2



FIG. 3

FIG. 2. Photomicrograph of gabbro, showing on the right a large crystal of labradorite in extinction position with blebs of stilbite (white); at the center a lath of oligoclase which has more successfully resisted paulopost action; near the center myrmekitic intergrowth of plagioclase (dark) and stilbite with larger masses of stilbite (white) above and below; at upper left the basal section of a quartz crystal.  $\times 43$ .

FIG. 3. Photomicrograph of gabbro with a mass of stilbite at the center (white) and myrmekitic plagioclase and stilbite above it. Plagioclase is seen as rounded remnants and shreds in crystallographic continuity with each other and with the parent plagioclase crystal immediately above.  $\times 43$ .



the residual liquids have caused a partial resolution of plagioclase crystals, frequently producing irregular blebs and veinlets along fracture lines as shown in Fig. 2. It is likewise clear that much of the interstitial stilbite is not filling existing interspaces between earlier crystals but occupies space made for it by active solution of plagioclase, a process which likewise produced a myrmekitic intergrowth of plagioclase, stilbite, and sometimes quartz. Again this process of resolution has been so complete that along a border irregular shreds having simultaneous extinction are the only remaining evidence that feldspar has been displaced (see Fig. 3). This process affects all of the plagioclase but is selective in that the more calcic varieties suffer most. Other minerals likewise have yielded to this action. Olivine is replaced wholly or in part by serpentine, especially the brown variety bowlingite. Augite, pigeonite, and other mafic minerals occasionally show a border of chloritic alteration products.

Because of the very small size of the Camas Land stilbite crystals no chemical analyses were made but comprehensive analyses of this mineral by Winchell indicate that there is little variation in composition and that the precise formula is nearly  $\text{Na}_3\text{Ca}_9\text{Al}_{21}\text{Si}_{69}\text{O}_{160} \cdot 60\text{H}_2\text{O}$ .<sup>4</sup> Since most of the plagioclase has a composition which may be designated as calcic labradorite it is evident that the zeolite partially replacing it is, except for the water of hydration and additional silica, similar in composition to the replaced labradorite. This, coupled with the fact that quartz often occurs with the stilbite but of earlier generation indicates the residual solutions to have been of siliceous and aqueous content. The occurrence of calcite suggests that carbon dioxide may likewise have had a part in the reaction and zeolitization. These residual solutions of the last stage of magmatic activity were, until arrested by cooling, actively dissolving the plagioclase by penetrating along cracks and contemporaneously depositing quartz and stilbite.

<sup>4</sup> Winchell, A. N., A new theory of the composition of the zeolites, Part III: *Am. Mineralogist*, Vol. 10, p. 151, 1925.

# VEIN-LIKE MASSES OF PYRRHOTITE IN CHALCOPYRITE FROM THE WAITE-ACKERMAN-MONTGOMERY MINE, QUEBEC<sup>1</sup>

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Recently in the study of polished sections, certain masses have been described as residual veinlets,<sup>2</sup> but it is found that when some of these are critically studied, and attention directed to crystallographic units and grain boundaries, they possess characteristics indicating that they are not residual veinlets.

According to a publication on the geology of the Waite-Ackerman-Montgomery Mine; "Residual veinlets of pyrrhotite are common in chalcopyrite."<sup>3</sup> No proof is given for this statement. On a priori grounds, the possibilities are that these pyrrhotite masses are either pre-wall, that is residuals, or contemporaneous ex-solved material from chalcopyrite, or post-wall true veinlets. The results of a detailed examination indicate, however, that they consist of pyrrhotite which has been formed by ex-solution from the chalcopyrite.

While with the Geological Survey of Canada in 1931, the author collected some ore specimens from the above named mine. The most common minerals present are chalcopyrite, sphalerite and pyrrhotite. The characteristics of and the relationships between the chalcopyrite and certain masses of pyrrhotite will be discussed in this paper.

The pyrrhotite occurs as curving hairlike areas, which may be described as resembling either wisps or vermicules (Fig. 1). These pinch and swell from being extremely fine to widths of 0.007 mm., and vary in length from 0.08 mm. to 0.5 mm. They have smooth boundaries against the surrounding mineral which is always chalcopyrite. The wisps may occur singly or as three distinct units radiating from a common centre.

<sup>1</sup> Published with the permission of the Director, Geological Survey of Canada, Department of Mines, Ottawa. Presented before *The Mineralogical Society of America*, Cambridge, Mass., December 29, 1932.

<sup>2</sup> Peale, Rodgers, *The Geology of the Waite-Ackerman-Montgomery Ore Deposit*: *Trans. Can. Inst. Min. and Met.*, **34**, p. 208, 1931.

<sup>3</sup> *Loc. cit.*

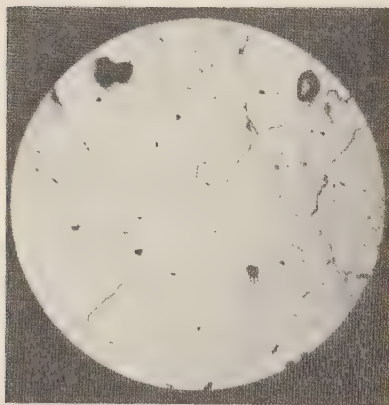


FIG. 1. Polished section of chalcopyrite containing vermicules or wisps of pyrrhotite; individual grains or crystals are not visible on this unetched surface.  $\times 24$ .

Without exception these pyrrhotite vermicules occur as thin sinuous areas of single crystals. That these are single pyrrhotite crystals is clearly shown by their behavior under polarized light. Any one wisp extinguishes uniformly and instantly along its entire length; that is, there is no variation in anisotropic colors or in extinction angle, all of which show that each pyrrhotite vermicule is a single crystal. This unity of crystallization is a property which frequently characterizes the minor constituent in accepted unmixing textures. It is to be observed here that, had the process been one of replacement of pyrrhotite by chalcopyrite to form residual veins, at least some of these vermicules should have consisted of more than a single crystal because the grain size of the coarsest pyrrhotite is only about one half the average length of the vermicules. On the other hand, had the grain size been sufficiently large it would appear most fortuitous indeed that no crystal boundaries are found in any one elongated area. For comparison specimens have been examined from other localities showing typical veins of pyrrhotite and of other minerals, and it was found that in every case the vein consisted of several crystals diversely oriented. In no case was a vein found to be composed of only one crystal, although it is possible that some may exist. It is obvious, then, that these vermicules differ also from later introduced material.

Not only are these vermicules developed as single crystals, but they are found only between crystals of chalcopyrite (Fig. 2).



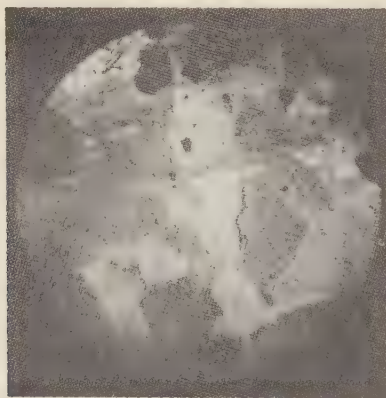


FIG. 2. Etched surface of chalcopyrite showing boundaries of the chalcopyrite crystals and the intra-crystalline nature of the pyrrhotite vermicules.  $\times 24$ .

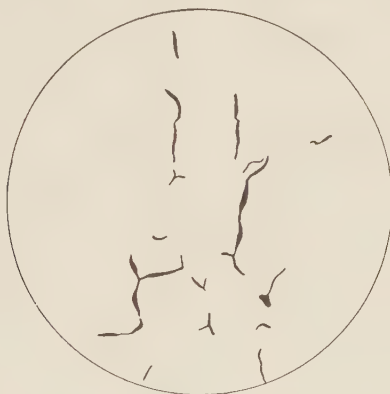


FIG. 3. Outline sketch of Fig. 2 showing location of the pyrrhotite vermicules. The thickness of the vermicules has been exaggerated slightly in the sketching.  $\times 24$ .

Under polarized light the crystal structure of the chalcopyrite is shown by the various anisotropic colors possessed by the different crystals, and by the interruption of the twinning lamellae at crystal boundaries. This structure is still more clearly shown when the chalcopyrite is etched with an acidic potassium dichromate solution.<sup>4</sup> The most interesting fact revealed by the crystal structure

<sup>4</sup> Schneiderhöhn, Hans, *Anleitung zur mikroskop. Bestimmung von Erzen*. 1922, pp. 116-117.

of the chalcopyrite is that the pyrrhotite wisps are always between the crystals of the former. The vermicules never cross-cut crystals of chalcopyrite as is a common habit of true veinlets. In some cases, a single vermicule may surround a chalcopyrite crystal on as many as three sides. Furthermore, even when three of these meet at a point, each one is seen to maintain very faithfully this inter-crystalline relationship to the chalcopyrite.

It is also significant that where vermicules join large rounded masses of early pyrrhotite there is always a crystallographic discontinuity at the edge of the rounded mass. Under polarized light, such a combination of vermicule and rounded mass always shows within itself different anisotropic colors and different extinction positions as between the single crystal of the wisp and the one or more crystals of the rounded mass. This feature has been observed and described before as an unmixing phenomenon. In some Frood ores described by W. H. Newhouse<sup>5</sup> such a crystallographic break obtains at the junction of large early pyrrhotite masses and later blades. The pyrrhotite blades in the Frood ore were held to be the result of unmixing. It is evident that the present described material shows analogous relations.

In addition to these features, the pyrrhotite masses fulfill three of the criteria<sup>6</sup> which are considered as suggesting ex-solution. First, the boundaries of the vermicules are smooth even when observed under very high magnifications; second, there is a total absence of these wisps outside of the chalcopyrite; and last, and most important, there is always a distinct narrowing of the vermicules where two or more join each other.

The chalcopyrite which is associated with this pyrrhotite is somewhat anomalous in that it is slightly more anisotropic than is usually the case. In addition, it shows very well developed twinning lamellae; the anisotropism is, however, not nearly as strong as that displayed by cubanite. The strong anisotropism may perhaps be explained by supposing that more iron is present in the space lattice than is usual in chalcopyrite. It may be suggested that all the excess pyrrhotite components have not been forced out of the chalcopyrite space lattice.

<sup>5</sup> Newhouse, W. H., A Pyrrhotite-Cubanite-Chalcopyrite Intergrowth from the Frood Mine, Sudbury, Ontario: *Am. Mineral.*, **16**, p. 337, 1931.

<sup>6</sup> Schwartz, G. M., Textures due to Unmixing of Solid Solutions: *Econ. Geol.*, **26**, pp. 761-762, 1931.

In summary, this would appear to be an unusual occurrence of pyrrhotite unmixing from chalcopyrite with no cubanite present. It might be noted in passing that a very careful study of crystal boundaries and of the crystallographic relationships between the different minerals present may be necessary before the origin of the veinlike masses may be determined.

The writer wishes to express his thanks to Professor W. H. Newhouse of the Department of Geology, Massachusetts Institute of Technology, for proposing the investigation and for valuable suggestions offered during its progress, and to the Geological Survey of Canada for permission to use the material collected while with them.

## STABILITY RELATIONS OF A COLORADO PISANITE (CUPRIAN MELANTERITE)

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Several specimens of pisanite, the isomorphous mixture of cupric and ferrous sulphates with 7 H<sub>2</sub>O, were collected in the Rico mining district in southwestern Colorado during the 1931 field season. Following Schaller's suggestion<sup>2</sup> the mineral should be called cuprian melanterite, as it contains more iron than copper. The rapid dehydration of the specimens after collection suggested a laboratory study of the relation between water content and humidity in this mineral. The results, which are recorded in this paper, have proved of considerable importance in the preservation of specimens and in the analytical determination of water of crystallization. The writer wishes to acknowledge his indebtedness to his colleague Mr. T. S. Lovering, for many valuable suggestions regarding the laboratory investigation, and to Mr. P. G. Nutting, also of the Geological Survey, for notes on the dehydration method adopted.

The cuprian melanterite was found only in one stope of the Wellington mine at Rico. It occurs as crusts on timbers or on the walls of a large body of pyrite which has almost completely re-

<sup>1</sup> Published by permission of the Director, U. S. Geological Survey, The Colorado State Geological Survey Board, and the Colorado Metal Mining Fund.

<sup>2</sup> Schaller, W. T., Adjectival ending of chemical elements used as modifiers to mineral names: *Am. Mineral.*, **15**, pp. 566-574, 1930.



placed a bed of limestone. The pyrite body contains considerable galena and sphalerite and a little chalcopyrite as the ore minerals. The principal post-mining oxidation products other than cuprian melanterite are gypsum, chalcantite, and a yellow sticky mud composed of one or more hydrous ferric sulphates. The waters in this part of the Wellington mine contain noteworthy amounts of ferric sulphate, copper sulphate and free sulphuric acid.

The cuprian melanterite, which ranges in color from pale delicate green, tinged with blue, to a darker bluish green, does not differ appreciably in its optical and other physical characteristics from those recorded in the literature for the mineral. The refractive indices as determined by Mr. Charles Milton of the U. S. Geological Survey are:  $\alpha = 1.473$ ,  $\beta = 1.478$ ,  $\gamma = 1.483$ ; with  $\gamma - \alpha = .010$ .

The results of an approximate analysis by Dr. R. C. Wells of the Survey are given in table 1. The material when analyzed by him had adsorbed deliquescent water and part of the iron had oxidized. Later when the laboratory study outlined below had furnished means for closely controlling the water content, Mr. F. G. Hills of the Colorado School of Mines Experimental Plant kindly made a partial analysis of material known to contain the seven molecules of water required by the formula of the mineral. Unfortunately it was then impossible to obtain samples which had not undergone slight oxidation since collection.

TABLE 1

Cuprian melanterite (pisanite), Rico, Colo. Analysts: 1, R. C. Wells; 2, F. G. Hills		
	1	2
CuO	7.2	$\left. \begin{array}{l} 16.98 \\ 1.18 \end{array} \right\} \approx 18.04 \text{ FeO}$
FeO	18.0	
Fe <sub>2</sub> O <sub>3</sub>	..	
ZnO	1.3	
MgO	tr.	
SO <sub>3</sub>	28.6*	
H <sub>2</sub> O	45.0**	44.3
	100.0	

\* Calculated.

\*\* Calculated, and eliminating 3.8% excess water in material as weighed.

This corresponds approximately to the formula:  $2 [\text{FeSO}_4 \cdot 7\text{H}_2\text{O}]$   
 $[(\text{Cu}, \text{Zn})\text{SO}_4 \cdot 7\text{H}_2\text{O}]$ .

## STABILITY RELATIONS

A laboratory study of the stability relations of the mineral was made in order to determine the humidity conditions under which it will remain stable. The results of that study lead to conclusions that seem important in any study of the hydrous sulphate minerals.

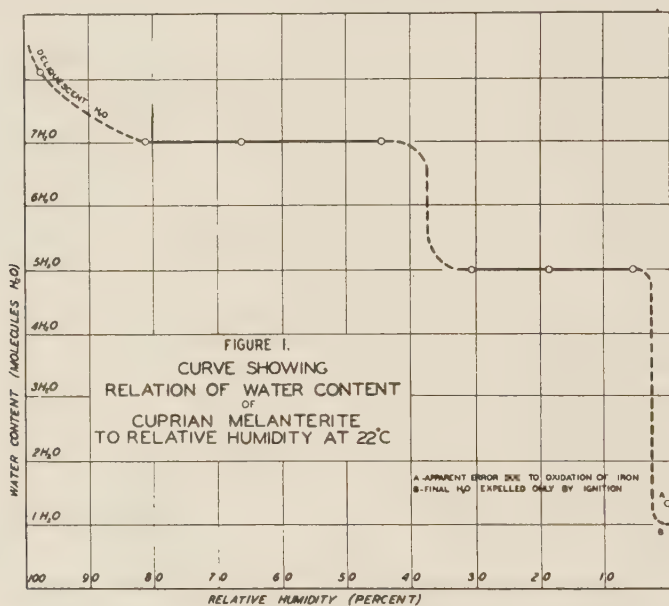
Briefly, the method of attack was to allow weighed samples to come to equilibrium in atmospheres of known humidity. The relative humidities over various concentrations of sulphuric acid were calculated from standard tables of chemical and physical constants and powdered samples of the mineral were placed in desiccators containing these solutions of acid. When the samples had come to equilibrium, as shown by the accordance of several daily weighings, they were then placed over acid corresponding to a slightly lower humidity. Abrupt changes in weight were found to

TABLE 2

H <sub>2</sub> SO <sub>4</sub> Solution	Sp. Gr. @ 22 deg. C.	Relative Humidity (%)*	Sample Weight grams	Loss in Weight	Remarks
1	1.040	97.8	See remarks		Increased in wt. and finally dissolved.
2	1.180	81.3	1.000	—	Blue green, corresponds to 7H <sub>2</sub> O.
3	1.260	66.6	0.998	—	No change.
4	1.357	44.6	0.998	—	No change.
5	1.425	30.7	0.865	0.135	Greenish white, loss of 2H <sub>2</sub> O to give pentahydrate.
6	1.490	18.7	0.865	—	No change.
7	1.598	5.3	0.865	—	No change.
8	1.835	near 0	0.646	0.354	Dark gray; loss of 4 more H <sub>2</sub> O to give monohydrate.

$$* \text{ Relative humidity} = \frac{\text{Partial pressure of water vapor in air over H}_2\text{SO}_4}{\text{Partial pressure of water vapor in air over H}_2\text{O}} @ 22^\circ\text{C.}$$

occur at three points, the losses in weight corresponding closely to the removal of a definite number of molecules of water of crystallization. Six of the seven molecules of water were thus removed at a constant temperature of 22° C., but the seventh can be removed only at a much higher temperature. The experimental results are tabulated in Table 2 and shown graphically in Fig. 1.



As shown in Fig. 1 the broken curved lines do not represent a gradual loss of water over a considerable humidity range, and it seems probable that the changes shown occur at definite points corresponding to the vapor pressures of the several hydrates at 22° but at present the data are insufficient to allow the plotting of a sharper curve. The temperature varied within limits of about 5° C., but no weighings were made until the temperature had remained at approximately 22° for several hours.

The loss in weight over concentrated acid was slightly less than that which would correspond with the removal of six molecules of water. This apparent discrepancy is accounted for by the fact that part of the iron was oxidized during the process of desiccation, with consequent increase in weight. Mr. F. G. Hills found that approximately 25% of the iron in the dehydrated material was in the ferric



condition. Since reducing atmospheres are seldom found in nature it was not thought worthwhile to repeat the experiments under conditions which would prevent the oxidation of ferrous iron.

Several conclusions of interest to those charged with the analysis or preservation of specimens of the type described here may be drawn from the experimental results. Although the vapor pressures of the various hydrates of pure cupric or ferrous sulphates have been determined by a number of workers, no data for the equilibrium conditions of the mixed sulphates have been available heretofore. It is obvious that in order to preserve specimens of the type described above they must be kept in an atmosphere of between 50% and 80% relative humidity. U. S. Weather Bureau records show a surprisingly large daily variation in relative humidities, so that even though the average monthly or yearly humidity in any locality falls well within the limits specified, it is unwise to place much reliance on natural conditions for preservation of rare or valuable specimens. It would seem far better to place them in desiccators over some medium that would insure an atmosphere of the correct humidity over long periods of time. Sulphuric acid is easy to use and to control, but there is some danger of spillage, and evaporation of water will produce marked changes in humidity. The International Critical Tables<sup>3</sup> list a number of solids, whose saturated solutions in contact with the solid phase give close control of the humidity of the surrounding atmosphere.

Chemical analyses and determination of the optical properties of the hydrous sulphates would be of much more value were they made on samples in equilibrium with atmospheres whose relative humidity was known. Certainly no chemist who has occasion to work with such compounds can fail to recognize the value of analyzing material known to contain the "correct" number of molecules of water of crystallization. It is probable, too, that the tendency for ferrous iron to oxidize is stronger when the mineral is not in equilibrium with the atmosphere. Whether small differences in the amount of water of crystallization affect the optical properties of the sulphate minerals has not been determined, but the possibility of such effects is evident.

The geological importance of the relationship between water of crystallization and humidity is not yet entirely clear, but it seems probable that humidity controls in part the formation of the vari-

<sup>3</sup> International Critical Tables, Vol. I, pp. 67-68, *New York*, 1926.

ous species, as well as their stability after they are formed. A detailed study of the stability relations of the isomorphous series of copper, iron and zinc sulphates is now in progress, and the writer hopes to present data for the equilibrium relations of several members of the series at a later date.

## NOTES AND NEWS

### OCCURRENCE OF RUBIDIUM, GALLIUM AND THALLIUM IN LEPIDOLITE FROM PALA, CALIFORNIA

T. G. KENNARD AND A. I. RAMBO, *Claremont Colleges*.

Lepidolite from the Stewart mine at Pala, Calif., has been reported to contain about 0.3%  $\text{Cs}_2\text{O}$ .<sup>1</sup> Various lepidolites from this locality have been analyzed by Schaller, who, however, makes no mention of rubidium, cesium, gallium or thallium.<sup>2</sup> It is interesting to note that the element rubidium was originally discovered in a lepidolite from Saxony, by Bunsen.<sup>3</sup> Traces of gallium have been reported in lepidolite from San Bernardino County, Calif.<sup>4,5</sup>

A sample of grayish white lepidolite obtained from the Sickler mine at Pala was examined spectrographically, using an electric arc as the source of excitation of the spectra, which were photographed by means of a large Gaertner quartz spectrograph. Examination of the plate revealed spectral lines due to rubidium, cesium, gallium and thallium, as well as other elements. (Fig. 1.)

Rubidium chloride and cesium chloride were subsequently prepared from the lepidolite. The method in brief consisted of decomposing sintered lepidolite with sulphuric acid, concentrating the rubidium and cesium first as alums and then as chlorostannates. The rubidium and cesium were freed from potassium by fractionally recrystallizing as rubidium cesium chlorostannate, and were

<sup>1</sup> R. M. Santmyers, *Information Circular No. 6215*, U. S. Bureau of Mines, 1930.

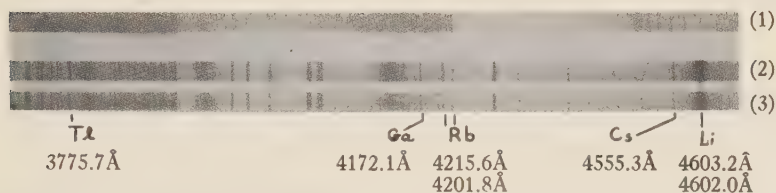
<sup>2</sup> W. T. Schaller, *U. S. Geol. Surv., Bull.* **419**, p. 287.

<sup>3</sup> R. Bunsen, *Ber. Akad. Berlin*, 273, 1861, *Chem. News*, **3**, 357, 1861; *Phil. Mag.*, [4], **22**, 55, 1861.

<sup>4</sup> J. Papish and D. A. Holt, *J. Phys. Chem.*, **32**, 142, 1928.

<sup>5</sup> Since submitting this article for publication, there has come to the author's attention an article by W. J. Schiefflen and T. W. Capon (*J. Soc. Chem. Ind.*, **27**, 549, 1908), in which it is stated that during the preparation of lithia from Pala lepidolite, rubidium and cesium alums were obtained in one stage of the process. Schiefflen and Capon do not state, however, that rubidium and cesium occurred in the lepidolite.

separated from each other by crystallizing as rubidium bitartrate and cesium antimony chloride, respectively, from which rubidium chloride and cesium chloride were prepared. The spectrographic examination and chemical procedure are described by us more fully elsewhere.<sup>6</sup>



(1) Spectrum of graphite electrodes.  
(2, 3) Spectrum of Lepidolite. FIG. 1

The quantities of rubidium chloride and cesium chloride obtained correspond to 0.67%  $\text{Rb}_2\text{O}$  and 0.16%  $\text{Cs}_2\text{O}$  in the raw lepidolite. The actual cesium and rubidium content of the lepidolite was presumably slightly higher, since traces of these elements were lost in the process of extraction and purification. The gallium and thallium were present in traces only, the amount being estimated from the intensities of their spectral lines.

The occurrence of the rubidium, cesium, gallium and thallium in the lepidolite may be explained on the basis of isomorphous replacement, in which the rubidium, cesium and thallium replace part of the potassium, and the gallium replaces part of the aluminum. Similar replacement of potassium by rubidium and thallium, and of aluminum by gallium, has been described for various minerals, other than lepidolite, from the Katzenbuckel Mts. in Odenwald.<sup>7</sup>

<sup>6</sup> Article submitted to *Ind. Eng. Chem.*

<sup>7</sup> F. Schröder, *Neues Jahrb. Mineral., Geol., Abt. A*, **63**, 215, 1931.

#### THE CRYSTAL STRUCTURE OF POTASSIUM DITHIONATE, $\text{K}_2\text{S}_2\text{O}_6$

MAURICE L. HUGGINS, (*Johnston Scholar in Chemistry*), *Johns Hopkins University*.

Because of criticisms<sup>1,2</sup> of some of the results reported in a paper having the above title by Huggins and Frank<sup>3</sup> the writer has re-

<sup>1</sup> Hägg, *Zeit. Krist.*, (A) **83**, 265, 1932.

<sup>2</sup> Helwig, *Zeit. Krist.*, (A), **83**, 485, 1932.

<sup>3</sup> Huggins and Frank, *Am. Mineral.*, **16**, 580, 1931.



cently carefully gone over the work reported in that paper. Nothing seems to be wrong with the structure analysis as originally carried out but two serious errors were made in the final preparation of the paper. The writer greatly regrets these errors and takes the full blame for them.

In the first place, the potassium atom parameters given in Table 4 were not those deduced from the data and used in the calculation of structure factors and interatomic distances. They should have been given as  $u_k = 0.375$  and  $u_k' = 0.690$ . Also the oxygen atoms in one of the  $S_2O_6^{--}$  groups were incorrectly placed in Figure 6. The positions shown agree neither with the symmetry of the space group nor with the atomic coordinates listed in Table 4. A correctly drawn figure is now given.

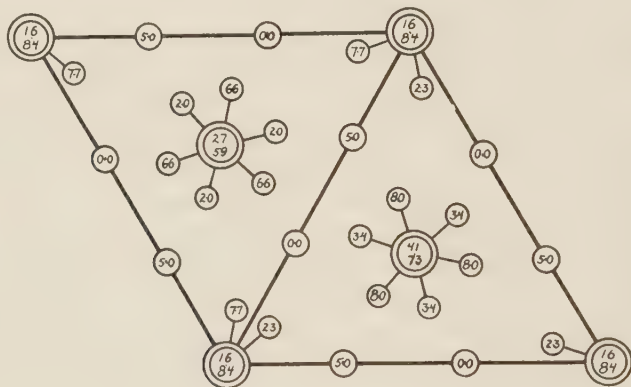


FIG. 1. (Replacing Fig. 6 in the paper by Huggins and Frank.) The distribution of atomic centers in the unit cell of  $K_2S_2O_6$ . Small circles represent oxygen, large circles potassium and large double circles pairs of sulfur atoms. The figures are the  $z$  coordinates, in hundredths of the unit distance.

Helwig's criticism that "certain planes which from symmetry considerations should give equal intensities of reflection are represented in the tables by  $F$  values which differ considerably" is easily explained. These  $F$  values were calculated (as stated in our paper) without making any correction for the differences in absorption and in the number of cooperating planes due to differences in length of path through the crystal for different reflections. Although these corrections could not be quantitatively made they should vary regularly along each of the rows (vertical, horizontal or

diagonal) in Table 5, in which the positions of the indices correspond roughly to the relative positions of the reflections on the photograph and of the points of the reciprocal lattice projection made from it. The omitted corrections would be very different for reflections of rays having very different path lengths through the crystal, as in the cases of the pairs of reflections mentioned by Helwig.

Helwig's  $F_{\text{obs}}$  values are in good agreement with ours but include more reflections. Calculations of  $F$  values for all of the reflections listed by him (a) as made by him, using atomic  $F$  curves of unknown origin, (b) as made by me, using his parameter values and the atomic  $F$  curves given in Ref. 3, and (c) as made by me, using the Huggins and Frank parameter values, show practically the same measure of agreement with the  $F_{\text{obs}}$  values. The last however seems to be slightly the best.

Although Helwig implies that he used the concept of interatomic distances to deduce his parameter values, the latter lead to improbably small values (2.20 Å for one kind of potassium, 2.31 Å for the other) for the distances between each potassium center and the centers of two of its six oxygen neighbors. K-O distances in other crystals (See Ref. 3, p. 590) are always much larger. For this reason the writer believes that Helwig's values for the parameters cannot be correct.

The results of these structure factor and interatomic distance calculations are given in more detail in a paper being sent to the *Zeitschrift für Kristallographie*.

#### DIRECTORY OF AMERICAN AND CANADIAN MINERAL COLLECTIONS

SAMUEL G. GORDON, *Academy of Natural Sciences  
of Philadelphia.*

(Continued from page 418.)

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nue.

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WESTERN RESERVE UNIVERSITY, Geology  
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NORMAN	UNIVERSITY OF OKLAHOMA.

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## GETTYSBURG                      GETTYSBURG COLLEGE.

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## HARRISBURG                      STATE MUSEUM, in Capitol group of buildings.

A small mineral collection.

## HAVERFORD                      HAVERFORD COLLEGE.

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JOHN TALLIS.

A small local collection.

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WASHINGTON	WASHINGTON AND JEFFERSON COLLEGE.
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	Library Building. Open weekdays.

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WILKES-BARRE	WYOMING HISTORICAL AND GEOLOGICAL SOCIETY.
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Frances Dorrance, Director. A general collection of several thousand specimens.

WILLOW GROVE	JOHN VANARTSDALEN.
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A local collection.

#### RHODE ISLAND

KINGSTON	RHODE ISLAND STATE COLLEGE.
PROVIDENCE	BROWN UNIVERSITY.
	PARK MUSEUM (Roger Williams Park).

The H. Anthony Dyer Collection.

WESTERLY	PUBLIC LIBRARY.
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A general collection, including the cabinet of Charles Hyde Denison. Two-hundred specimens are permanently displayed. Open daily from 9 A.M. to 9 P.M.

#### SOUTH CAROLINA

CHARLESTON	CHARLESTON MUSEUM.
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Has the Daniel S. Martin Collection (in part), and a collection of phosphates assembled by Charles U. Shepard.

COLLEGE OF CHARLESTON.  
SOUTH CAROLINA MILITARY ACADEMY.

CLEMSON	CLEMSON COLLEGE.
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CLINTON	THORNWELL MUSEUM.
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COLUMBIA	UNIVERSITY OF SOUTH CAROLINA, LeConte College.
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A general collection of 7000 specimens, including that of the State Geological Survey, the Thomas Cooper Collection (which received rough treatment dur-



ing the Civil War), the small Louis R. Gibbes collection (in part), and some of the meteorite material of C. U. Shepard. The collections may be seen daily from 9 A.M. to 5 P.M.

GREENVILLE      FURMAN COLLEGE.

Daniel S. Martin Collection, in part.

NEWBERRY      NEWBERRY COLLEGE.

ORANGEBURG      CLAFIN UNIVERSITY.

ROCK HILL      WINTHROP COLLEGE.

A general collection of 200 specimens. Miss Minnie Parker, Custodian. Open daily 9 to 5; Sundays 2 to 3.

SPARTANBURG      WOFFORD COLLEGE.

A small collection of minerals.

SOUTH DAKOTA

CUSTER      SCOTT ROSE QUARTZ COMPANY.

About 1000 local specimens.

RAPID CITY      SOUTH DAKOTA STATE SCHOOL OF MINES.

VERMILLION      UNIVERSITY OF SOUTH DAKOTA.

A general collection of 2000 specimens. Depository of the State Geological Survey.

YANKTON      YANKTON COLLEGE.

TENNESSEE

CLARKSVILLE      SOUTHWESTERN PRESBYTERIAN UNIVERSITY.

A large collection.

HARRIMAN      PUBLIC MUSEUM.

JACKSON      UNION UNIVERSITY.

KNOXVILLE      UNIVERSITY OF TENNESSEE.

Small teaching collections. Professor George M. Hall, Custodian.

LEBANON      CUMBERLAND UNIVERSITY.

MARYVILLE      MARYVILLE COLLEGE.

MEMPHIS      COSSITT LIBRARY MUSEUM ASSOCIATION.  
SOUTHWESTERN COLLEGE.

Has the Lardner Vanuxem collection, purchased by the earlier Masonic College of Clarksville, Tenn.

MILLIGAN      MILLIGAN COLLEGE.

JEFFERSON CITY      CARSON AND NEWMAN COLLEGE.

## NASHVILLE

## TENNESSEE DIVISION OF GEOLOGY.

State Geologist, Custodian. General state collections may be seen by visitors from 8.30 to 4.30 P.M. daily, and on Saturdays until 12 M.

## VANDERBILT UNIVERSITY, Garland Hall.

General teaching collections, 12,000 specimens. W. B. Jewell, Custodian.

## FISK UNIVERSITY.

## WALDEN UNIVERSITY.

## TEXAS

## ALPINE

## WEST TEXAS HISTORICAL AND SCIENTIFIC SOCIETY.

Sul Ross State Teachers College. Main Building. E. E. Townsend, Curator. A general and local collection of 600 specimens. Open Fridays 2 to 5 P.M., or by appointment.

## AUSTIN

## BUREAU OF ECONOMIC GEOLOGY. University of Texas: Museum Building.

A general collection of 4000 minerals and 2 meteorites. Open daily, 8.30 A.M. to 5 P.M.

## BROWNWOOD

## HOWARD PAYNE COLLEGE.

## DALLAS

## TEXAS MUSEUM OF NATURAL HISTORY.

## EL PASO

## EL PASO MUSEUM.

## PUBLIC LIBRARY.

Specimens are in storage for a future museum.

## UNIVERSITY OF TEXAS, Museum of the College of Mines and Metallurgy.

## HOUSTON

## HOUSTON MUSEUM OF NATURAL HISTORY.

C. L. Brock, Director. A general collection of 5000 specimens. Open 10 A.M. to 5 P.M. daily.

## SAN ANTONIO

## SCIENTIFIC SOCIETY OF SAN ANTONIO, Stevens Building.

## WITTE MUSEUM, Brackenbridge Park.

A general collection of 5000 specimens. Open daily from 9 A.M. to 6 P.M.

## SAN AUGUSTINE

## HORACE E. WOLCOTT

About 4000 minerals and gems, including many local specimens.

## WACO

## BAYLOR UNIVERSITY. Carroll Science Hall.

*(To be continued)*